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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods of Chemical Analysis of Red Lead¹

This standard is issued under the fixed designation D 49; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

^{ε1} NOTE—Keywords and References were added editorially in May 1996.

1. Scope

1.1 These test methods cover procedures for the chemical analysis of red lead having the approximate formula Pb_3O_4 (probably $PbO_2 \cdot 2PbO$).

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 50 Test Methods for Chemical Analysis of Yellow, Orange, Red, and Brown Pigments Containing Iron and Manganese²

D 215 Practice for Chemical Analysis of White Linseed Oil Paints³

D 280 Test Methods for Hygroscopic Moisture (and Other Matter Volatile Under the Test Conditions) in Pigments²

D 1193 Specification for Reagent Water⁴

D 1208 Test Methods for Common Properties of Certain Pigments²

D 1301 Test Methods for Chemical Analysis of White Lead Pigments²

D 1959 Test Method for Iodine Value of Drying Oils and Fatty Acids²

3. Treatment of Sample

3.1 If the pigment is lumpy or not finely ground, grind it to a fine powder and mix thoroughly. Large samples may be thoroughly mixed and a representative portion taken and powdered if lumpy or not finely ground. The sample in all cases shall be thoroughly mixed before taking portions for analysis. All samples shall be preserved in stoppered bottles or containers.

4. Purity of Reagents

4.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁵ Other grades may be used provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Type II of Specification D 1193.

5. Moisture

5.1 Determine moisture content with a 2-g specimen in accordance with Method A of Test Method D 280. The specimen is dried for 2 h at 105°C. The loss in weight is considered as moisture.

6. Organic Color

6.1 Boil 2 g of the sample with 25 mL of 95 % ethyl alcohol, let settle, decant the supernatant liquid; boil the residue with 25 mL of distilled water and decant as before; boil the residue with 25 mL of diluted NH_4OH (1 + 4) and again decant. Boil another 2-g portion of the sample with 25 mL of chloroform, let settle, and decant the supernatant liquid. If any one of the above solutions is colored, organic coloring matter is indicated. If the solutions remain colorless, organic colors are probably absent.

NOTE 1—If it is desired to test for organic colors resistant to the above reagents, the test procedures described in the following books may be used, taking into account the nature of the pigment involved (1,2,3).⁶

7. Total Lead and Insoluble Matter

7.1 Treat 1 g of the sample with 15 mL of HNO_3 (1 + 1) and sufficient H_2O_2 to dissolve all PbO_2 on warming. If any

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D 01.31 on Pigment Specifications.

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² *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vol 06.01.

⁴ *Annual Book of ASTM Standards*, Vol 11.01.

⁵ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopoeia and National Formulary*, U.S. Pharmacopoeial Convention, Inc. (USPC), Rockville, MD.

⁶ The boldface numbers in parentheses refer to a list of references at the end of these test methods.